

2021資源與環境學術研討會

日期：中華民國110年11月19日

時間：9:00~17:00

地點：大漢技術學院

主辦單位：大漢學校財團法人大漢技術學院

土木工程與環境資源管理系

協辦單位：國立成功大學奈米粉體科技研究中心

國立宜蘭大學環境工程學系

花蓮縣環境保護局

台泥企業集團

亞洲水泥股份有限公司花蓮製造廠

康廷工程顧問企業有限公司

日揚環境工程有限公司

亞太環境科技股份有限公司

祥威環境科技有限公司

新翊環境有限公司

春迪企業股份有限公司

承晏環境科技股份有限公司

長慧環境科技有限公司

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110 年 11 月 19 日 (星期五)

時 間	議 程			引言人 (主持人)
08：30 至 09：00	報到(土木館川堂)			
09：00 至 09：20	開 幕 貴賓致詞			王世忠 主任
09：20 至 10：20	專題演講 演講者：向性一 教授 演講題目：「廢棄鋰離子電池資源化技術」			胡紹華 教授
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10：50 至 12：00	Session I-1 (B215)	Session I-2 (B204)	Session I-3 (B219)	顏富士 向性一 陳智成
12：00 至 13：40	午 餐(B215、B204、B219)			
13：40 至 15：00	Session II-1 (B215)	Session II-2 (B204)	Session II-3 (B219)	余炳盛 蔡志達 何志軒
15：00 至 15：40	茶 敘(2F 走廊)			
15：40 至 17：00	Session III-1 (B215)	Session III-2 (B204)	Session III-3 (B219)	張章堂 林凱隆 謝哲隆
期待再重逢！				

2021 資源與環境學術研討會論文發表分組議程表

110 年 11 月 19 日 (星期五) 09:20~10:20

專題演講 B215

引言人：胡紹華 教授

演講者：向性一 教授（國立成功大學資源工程學系 系主任）

演講題目：廢棄鋰離子電池資源化技術」.....1

Session I-1 (10:50~12:00) B215

主持人：顏富士 教授（國立成功大學）

1. 顏富士、李孟穎、邱奕騰（國立成功大學 資源工程所）
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主持人：向性一 教授（國立成功大學）

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B219

主持人：陳智成 教授（遠東科技大學）

1. 張益誠、黃暉涵、余泰毅、李妍安、張宸翔、伍士右 (國立宜蘭大學 環境工程學系) 環境教育旅遊聊天機器人(Chatbot)雲端應用系統佈署研究芻議.....129
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B215

主持人：余炳盛 教授（國立臺北科技大學）

1. 陳建洲、江呈文、李韋皞 (國立臺北科技大學 資源所)
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B204

主持人：蔡志達 副研究員 (國立成功大學)

1. 張益誠、李妍安、蔡承儒、潘奉琪、李林伶、許展榮、余泰毅 (國立宜蘭大學 環境工程學系) 運用環境治理區塊鏈私鏈節點案例式導向雲端系統編程實例化 (instantiate)抽象技術特性之研究.....211
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主持人：何志軒 副教授 (大漢學校財團法人大漢技術學院)

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主持人：張章堂 教授 (國立宜蘭大學)

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主持人：林凱隆 教授 (國立宜蘭大學)

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主持人：謝哲隆 教授 (國立宜蘭大學)

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專題演講

演講者：向性一 教授

演講題目：「廢棄鋰離子電池資源化技術」



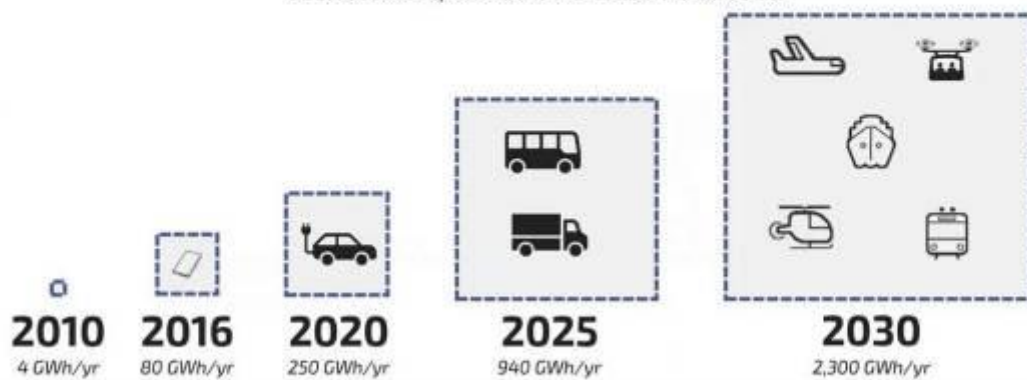
廢棄鋰離子電池資源化技術

向性一
資源工程學系
國立成功大學

EXPONENTIAL LI-ION BATTERY DEMAND GROWTH

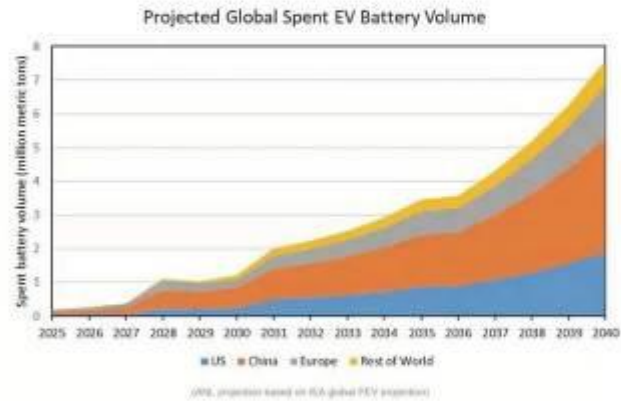
Lithium-ion battery demand globally has risen dramatically over the last 10 years, and is only beginning

Global demand depicted below with accurate relative scale



SETTING THE STAGE

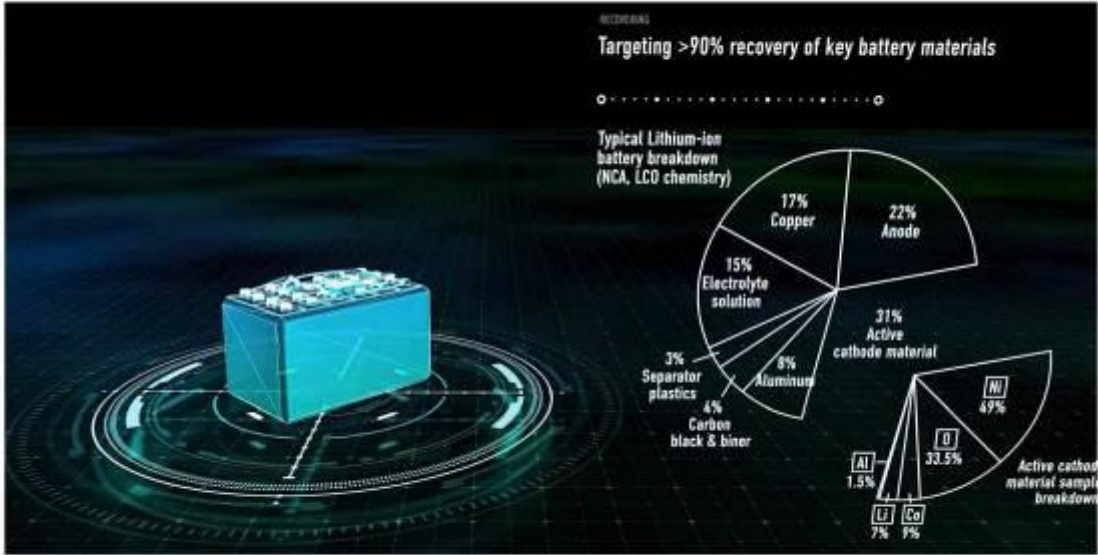
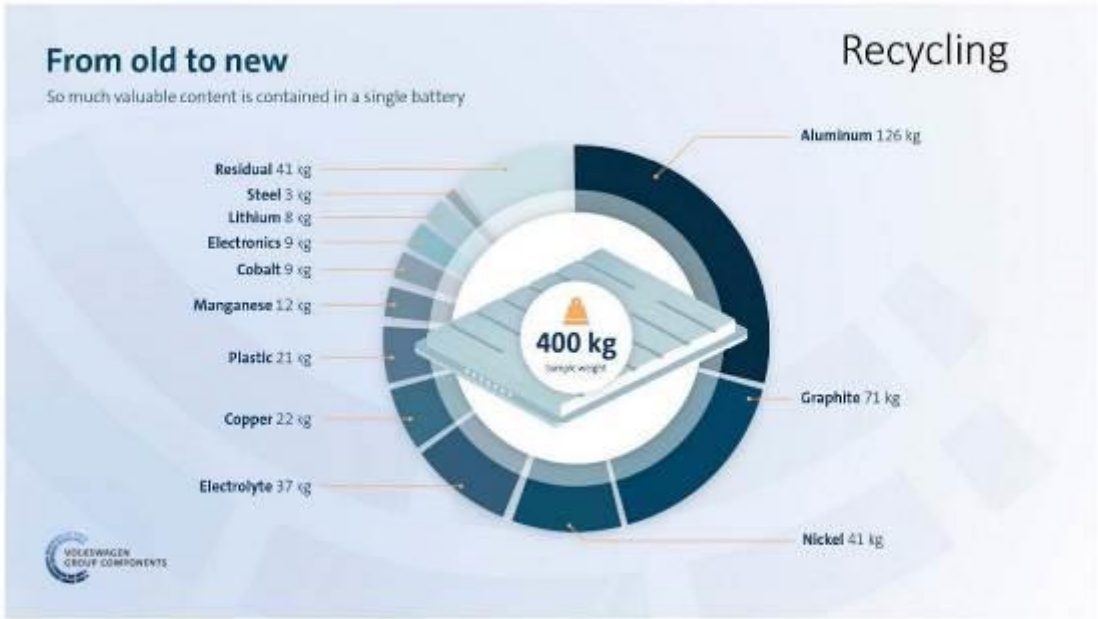
- An increase of lithium-ion batteries coming in electric vehicles (EV)
 - Consumer electronics collection is an issue
 - Stationary applications can be in remote locations
- Cannot meet EV material demand without recycling

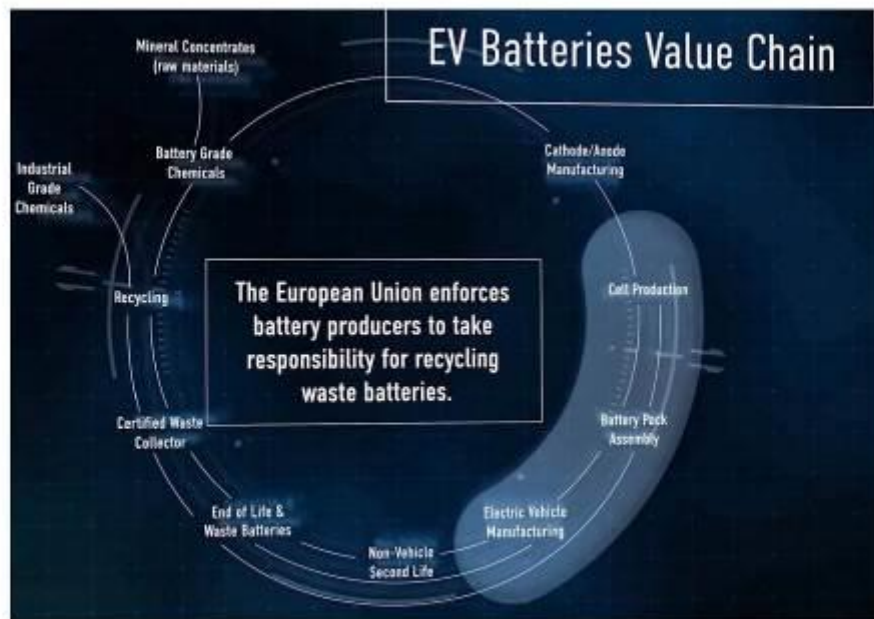


Types of LIBs in the market

	LCO - Lithium Cobalt Oxide	LFP - Lithium Iron Phosphate	LMO - Lithium Manganese Oxide	NCA - Lithium Nickel Cobalt Aluminium Oxide	NMC - Lithium Nickel Manganese Cobalt Oxide	LTO - Lithium Titanate
	LiCoO_2	LiFePO_4	LiMn_2O_4	LiNiCoAlO_2	LiNiMnCoO_2	Li_2TiO_3
Specific Energy, Wh/kg	150-200	90-120	100-150	200-260	150-220	50-80
Cycle Life	500-1,000	>2,000	300-700	500	1,000-2,000	3,000-7,000
Applications	Mobile phones, tablets, laptops, cameras	High load currents and endurance, E-buses, e.g. BYD	Power tools, medical devices, electric powertrains	Medical devices, industrial, electric powertrain, e.g. Tesla EV, Panasonic	E-bikes, medical devices, EV, e.g. Nissan LEAF, iMiEV, Volt	Uninterrupted power supply, electric powertrain

Source: Battery University







Li-cycle

- <https://youtu.be/-J8aMdxiZNI>
- https://youtu.be/Te8f_0k5X_g



Closed Loop Resource Recovery

Industry-leading technology enabling a closed-loop economy.



Logistics Management

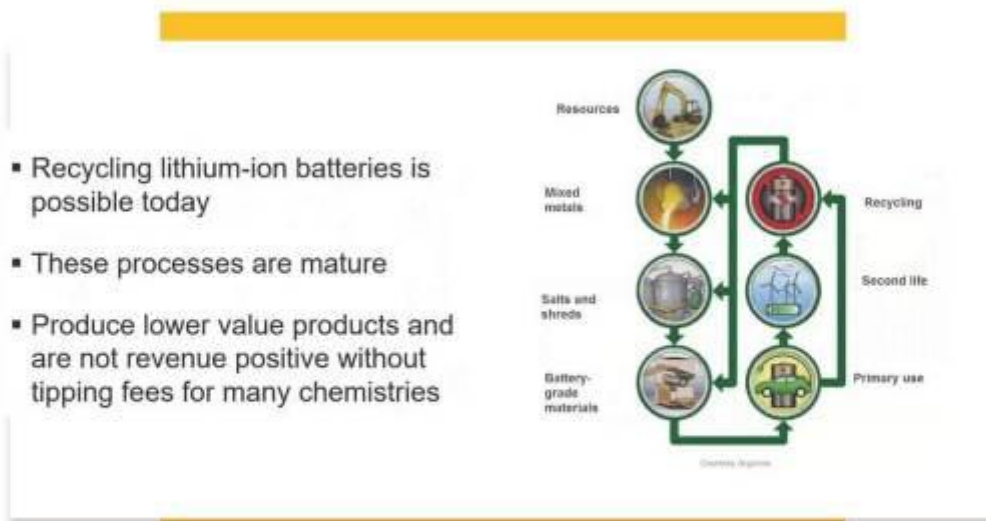
A reliable network of logistics partners to support customers in getting their batteries to our facilities.

Secure Destruction

A home for the secure destruction of materials containing IP-sensitive design information.

Add-on Services

Additional services customized to meet our customers' needs.



Current recycling process

Processing “black mass”

Comparison of main technologies

Pyrometallurgy



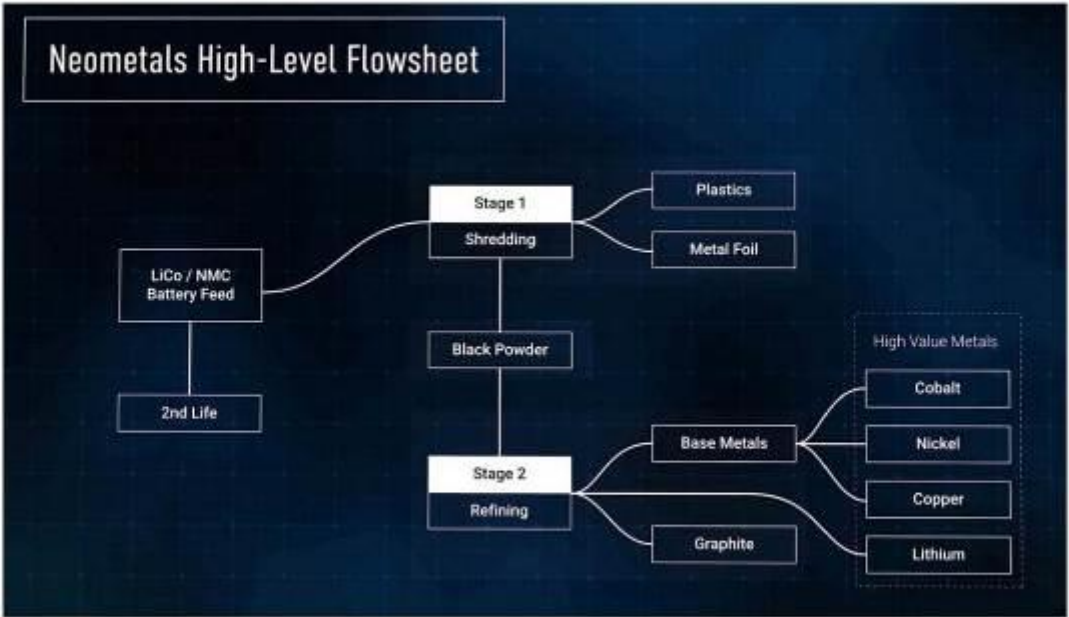
- ✓ High recovery rates for nickel, cobalt and copper
- ✓ Graphite and solvents burned, providing much of the process energy
- ✓ Mature technology
- ✗ High energy intensity (around 1,500°C) and CO₂ footprint
- ✗ Loss of lithium in slag – recovery from slag is expensive

Hydrometallurgy



- ✓ High recovery rates for cobalt, nickel and copper
- ✓ Lithium is recycled
- ✓ Option for manganese and graphite recycling
- ✓ Moderate temperature range
- ✗ High investment required
- ✗ Inflexible process
- ✗ High amounts of by-products, waste

Both technologies call for improvements towards lithium yield, by-products or investment cost.



RECYCLING SUPPLY CHAIN: INCUMBENT PROCESS

Incumbent recycling chain/processes

Disaggregated, inefficient, low recoveries, waste-oriented

~50% recovery rate



Spoke & Hub Technologies

A combination of mechanical safe size reduction and hydrometallurgical resource recovery specifically designed for lithium-ion battery recycling.

95% recovery

MECHANICAL SAFE SIZE REDUCTION

Spoke

All types of lithium-ion batteries are transformed from a charged state to an inert product in a safe and efficient manner.

HYDROMETALLURGICAL PROCESSING

Hub

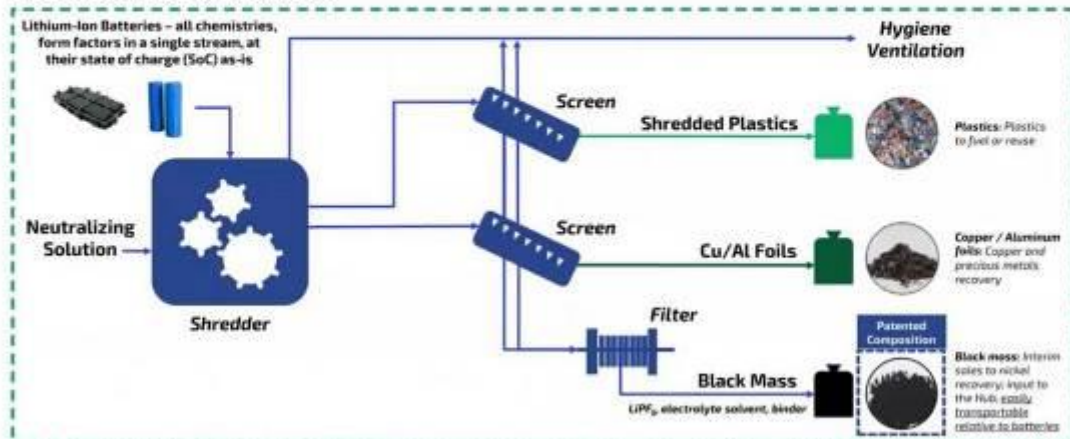
Cathode and anode materials are processed into battery grade end-products for reuse in lithium-ion battery production or other applications in the broader economy.

Spoke and Hub Technology of Li-Cycle



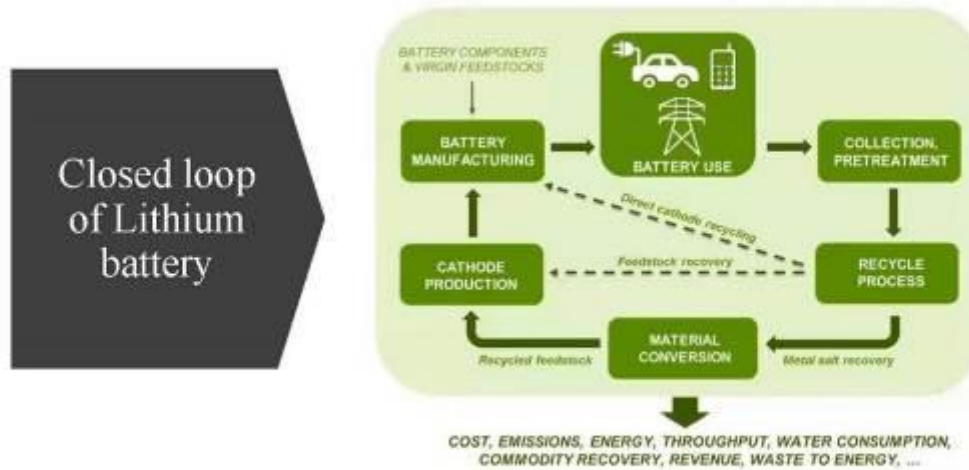
Spoke Technology

Patented Apparatus / System



Shredding-drying-sieving-magnetic separating-
electric separating 分選為塑料、鋁銅、電極粉末





“Black mass*” – a new resource

- Recycling of lithium-ion batteries (LIB) starts with mechanical operations (e.g., dismantling, shredding, sorting).
- This yields “black mass”.
- It mainly consists of the electrode active material.
- Black mass contains valuable Co, Ni, Mn, Li, but also carbon and many contaminants.

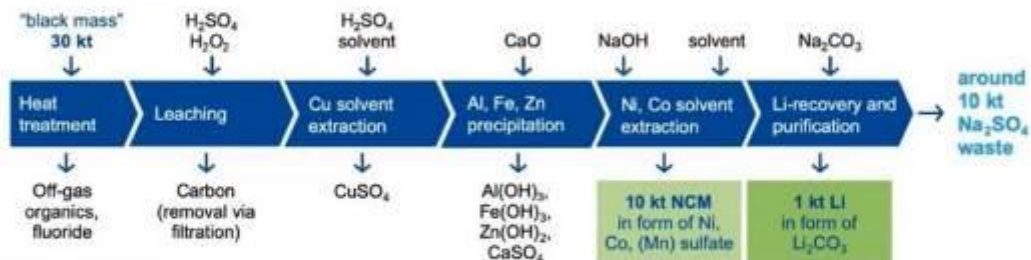
Chemical treatments are needed to extract the valuable metals from “black mass”.

Element	Content weight %**
Ni	20
Co	6
Mn	6
Li	3.5
C	40
F	3
P	0.5
Cu	2
Al	2
Fe	1
Zn	0.1
Ca	0.1



Deep dive hydrometallurgy

Illustrative process



Characteristics

- ✓ Well established processes in mining industry up to nickel and cobalt recovery to build on
- ✗ Inflexible process – lithium is always recycled at the end and comes as lithium carbonate only
- ✗ Significant amounts of sodium sulfate waste by-product

The process cuts CO_2 emissions by -25% vs. CAM* materials based on mining.

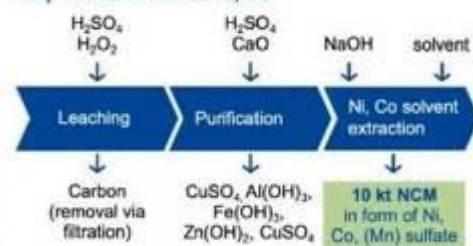
Step 1: Removal of lithium from "black mass"



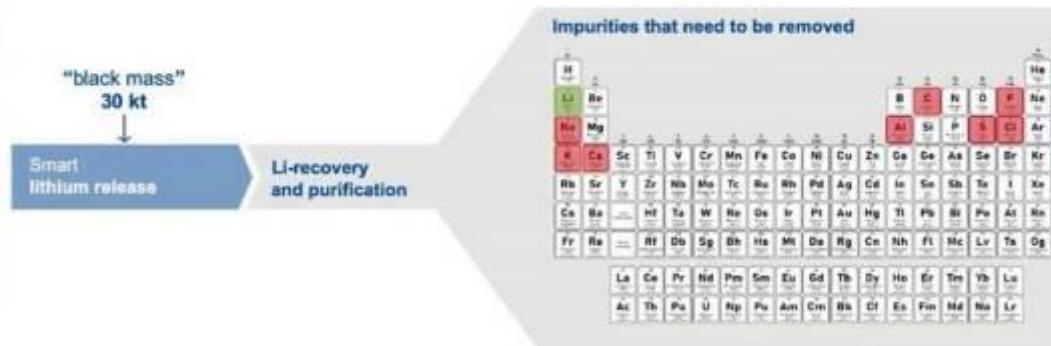
Benefits of LIOH first:

- ✓ avoids sodium sulfate by-product
- ✓ allows direct access to lithium hydroxide
- ✓ cuts investment cost in the value chain

Step 2: Extraction of Ni, Co

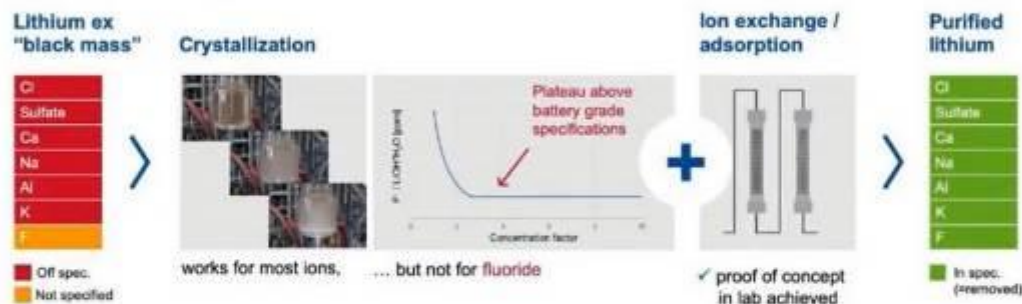


Deep dive lithium purification



New BASF purification scheme

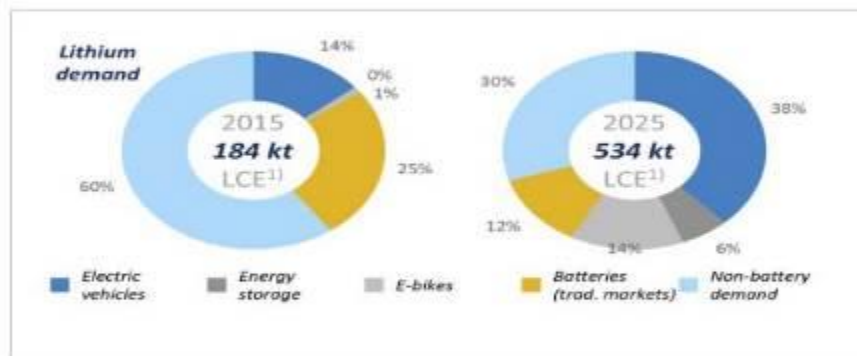
Recovering battery-grade lithium from "black mass"



Smart combination of purification technologies needed to extract battery-grade lithium from "black mass".

BASF

Lithium demand

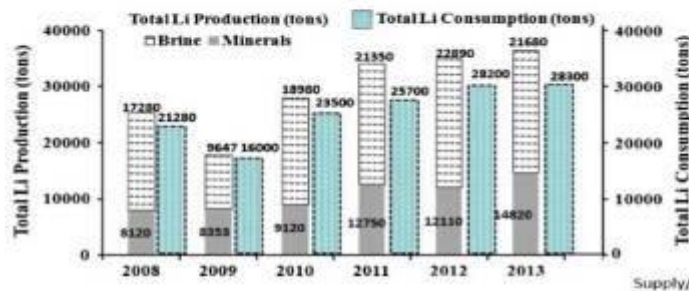


It suggests that the lithium demand strongly depends on the automotive electrification.

(Source: McKinsey, Bloomberg New Energy Finance, Deutsche Bank, Wealth Minerals)

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Demand and supply of lithium

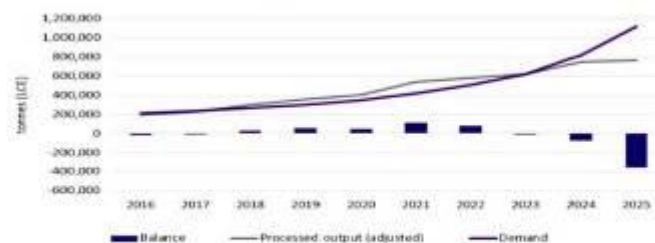


Lithium dissolved in brines accounts for more than 66 % of global lithium reserves.

Today, most of the lithium is produced (about 83 %) from brines.

Supply/Demand balance forecast

The deficiency of lithium carbonate will reach about 350 k tons.



1

Two main recovery resources of lithium

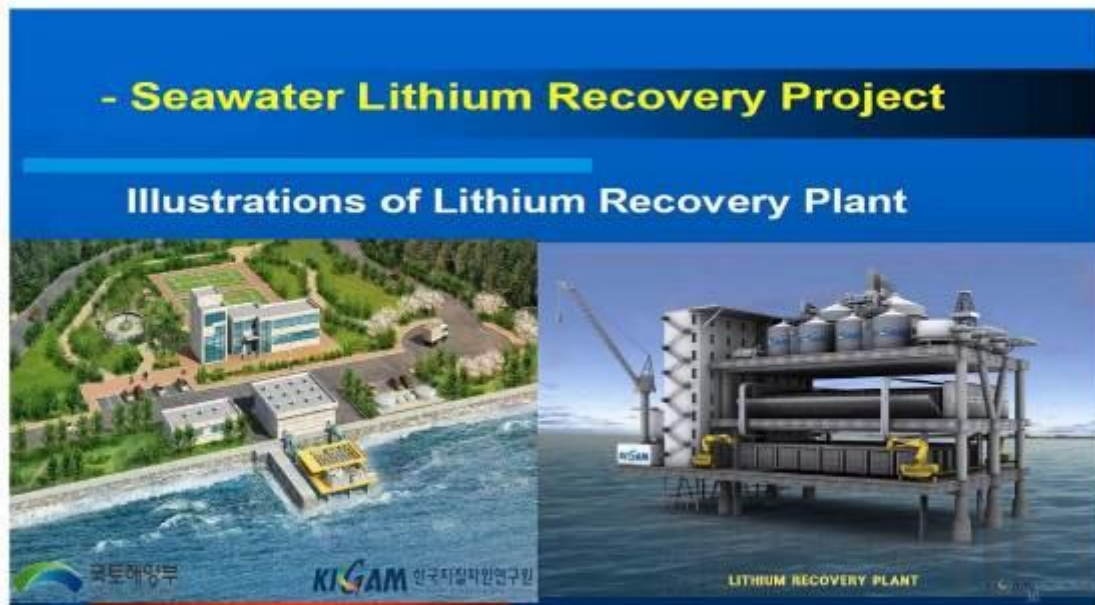
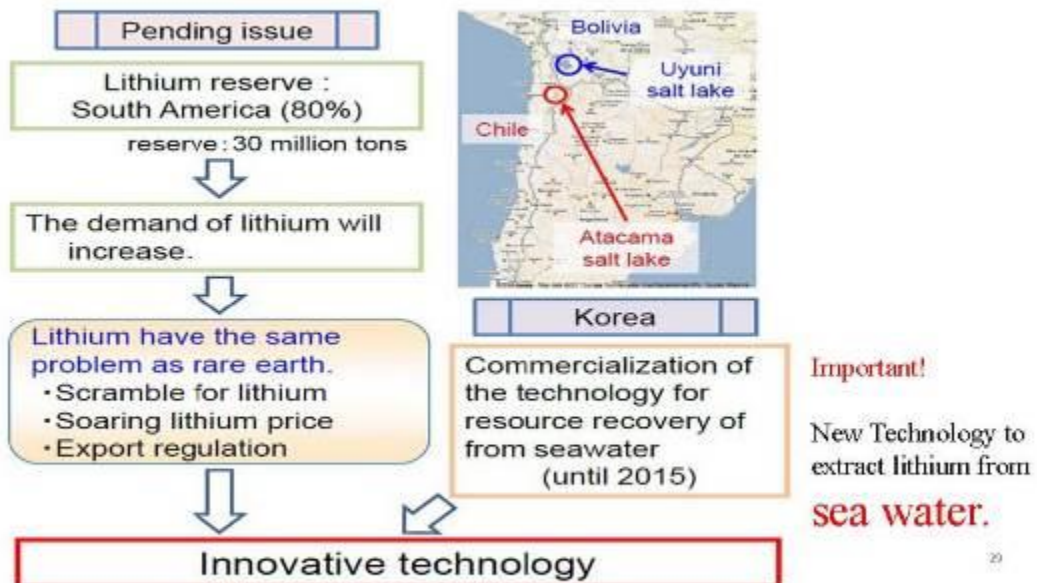
- Brine water
- Spent lithium battery

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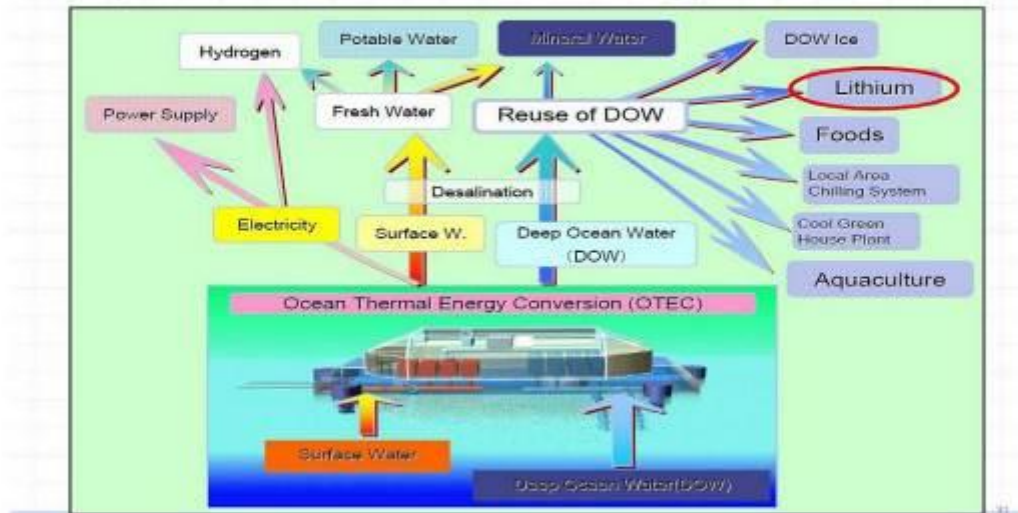
Sustainability: the choice



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Multiple Utilization of Ocean Energy and Resources



DIRECT RECYCLING



DIRECT RECYCLING

- Multiple processes investigated to mitigate risk
- Continual review of new project ideas
- End projects that are not showing promise in cost and performance
- These unit operations can benefit other recycling processes

Typical Direct Recycling Process Flow



Cathode recovery could enable economical recycling

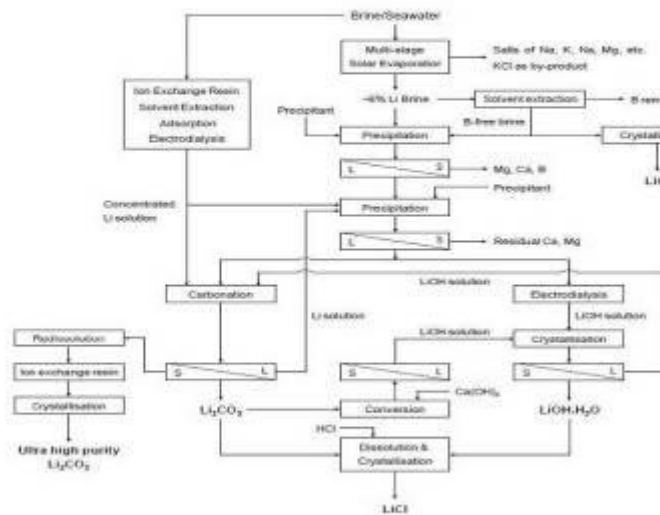
- Value of constituents for LiFePO_4 is low comparing with LiCoO_2 or $\text{Li}(\text{Co}, \text{Ni}, \text{Mn})\text{O}_2$, but cathode itself has high value.

Cathode	Price of Constituents (\$/lb)	Price of Cathode (\$/lb)
LiCoO_2	\$9.90	12.00 ^{3,4}
$\text{LiNi}_{0.3}\text{Co}_{0.3}\text{Mn}_{0.3}\text{O}_2$	\$6.10	\$8.80 ⁴
LiMnO_2	\$1.35	\$4.50 ²
LiFePO_4	\$0.75 ¹	\$9.10 ²

Spent LiFePO_4 battery will not be recycled to the constituents by using chemical or high temperature smelting processes due to the lack of economic benefits.

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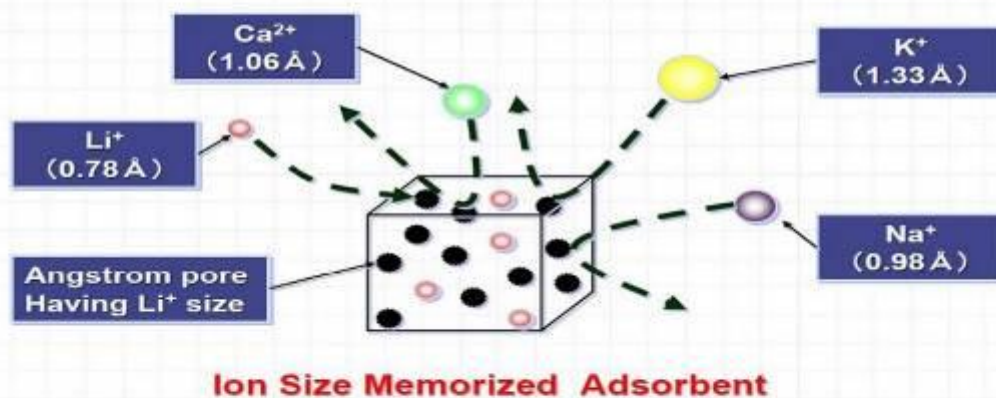
General flow sheet for production of lithium compounds from brines and seawater.



The traditional process is complex and needs many chemicals, which is detrimental to the environment.

Is there other simple and environmental friendly process to extract lithium from sea water?

Adsorbent Having High Selectivity for Lithium Ion Spinel Type $\text{Li}_x\text{Mn}_2\text{O}_4$



Synthesis of $\text{Li}_x\text{Mn}_y\text{O}_4$ and $\lambda\text{-MnO}_2$



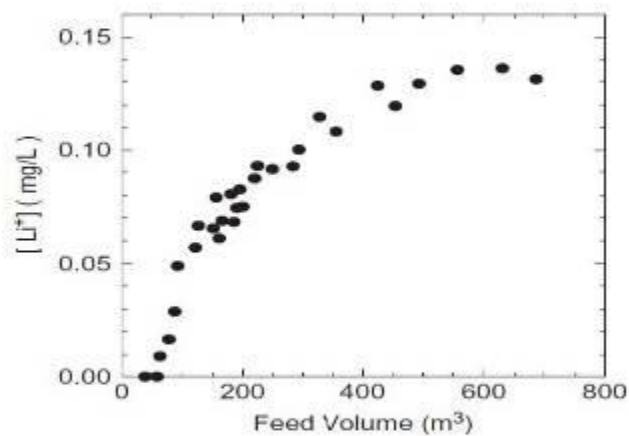
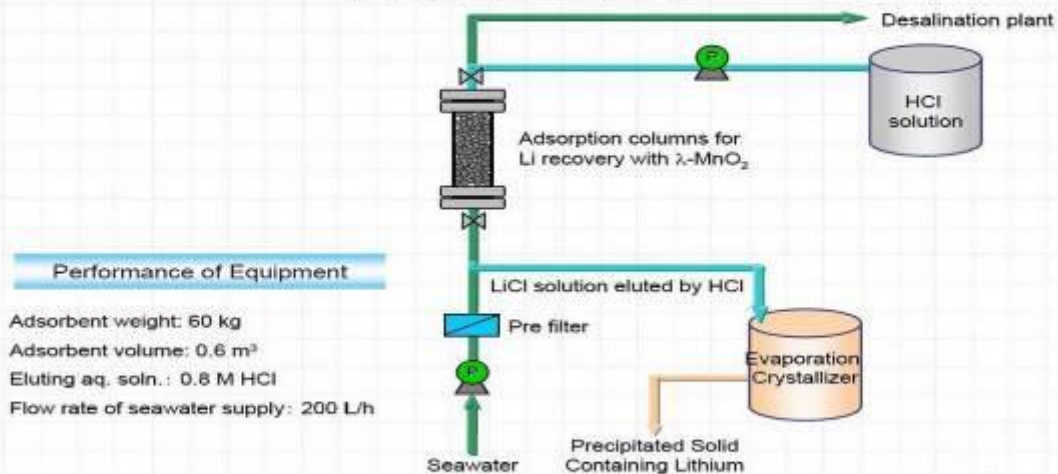
37

Granulation of Adsorbent for Column Packing



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Benchmark Plant of Lithium Recovery from Seawater in IOES



Lithium concentration profile after passing adsorption column.

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Ionic adsorption selectivity of MnO_2

Table 1. Metal composition of the eluted solution and the dried precipitate salt

Element	Eluted solution (mg/L)	Precipitated salt* (wt%)	Concentration efficiency from seawater
Li^+	160	33.3	11000
Na^+	236	20.4	0.26
K^+	92	3.3	0.94
Mg^{2+}	17	8.2	0.57
Ca^{2+}	176	13.4	4.11
Sr^{2+}	35	2.0	50
Mn^{2+}	234	19.4	—

*calculated as metal chlorides existing in the precipitates

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Problem of the ion sieve adsorption technology

- The main problem of the ion sieve adsorption technology: The adsorption capacity and rate became smaller and slower after granulation and adsorbent loss increased due to the degradation of the binder.

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Electrosorption of LiCl in different solvents by carbon nanotube film electrodes

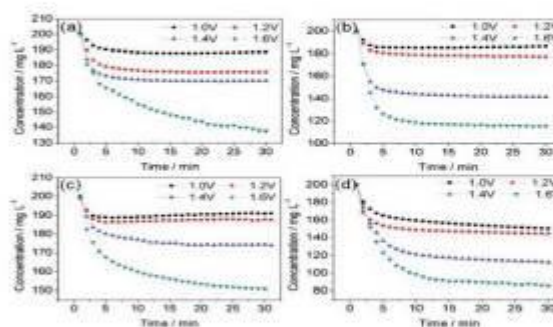
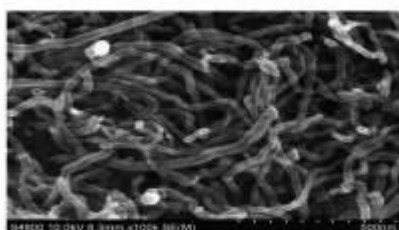
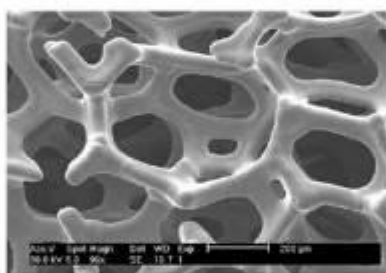


Fig. 2 Time-dependant UCl concentration variation in (a) water, (b) methanol, (c) ethanol and (d) DMF during the electrosorption at different voltages.

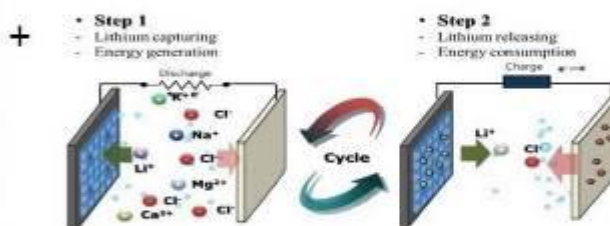
43

Purpose of research

Lithium ion sieve



Electro-adsorption



Methodology: The spent LiFePO_4 powder was coated onto the surface of titanium mesh by using electrophoresis to form a 3D nano-structured electrode, which can extract lithium from brine water via electro-adsorption process.

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Benefits of the combination of 3-D nanostructured lithium ion sieve and electrosorption

- Fast adsorption/desorption rate due to the combination of the 3D nanostructure, lithium ion sieve and electric field.
- No need of granulation.
- No need of membrane which is expensive, easily leads to biofouling and is easily permanently destroyed by certain solvents and colloidal solids.
- No need of washing acid, which is more environmentally friendly.

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Disassembly of spent LiFePO_4 battery



After discharging



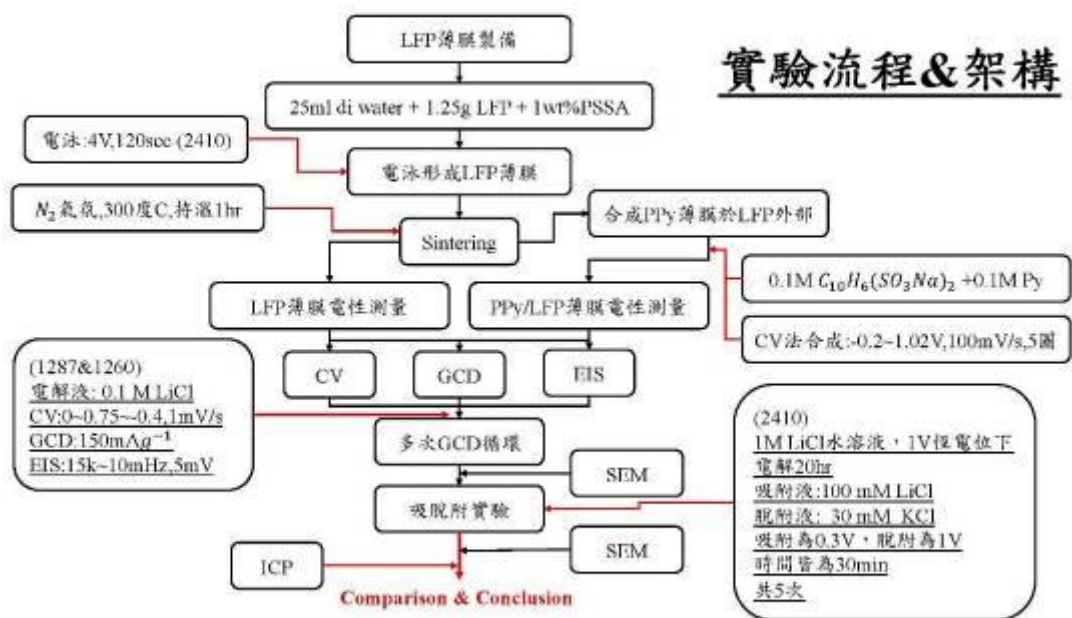
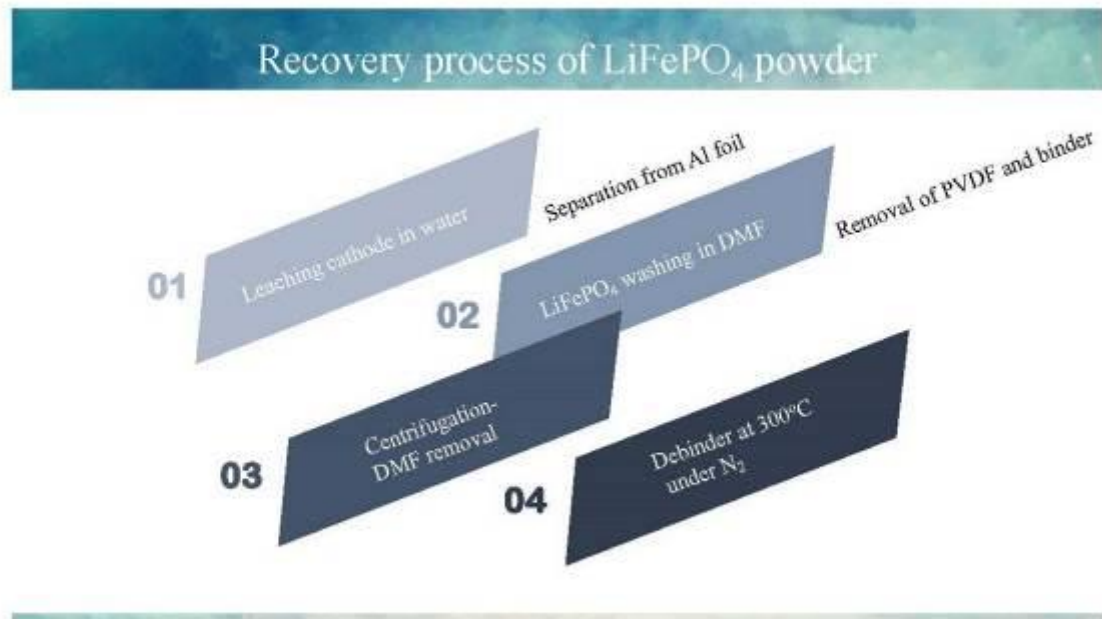
After disassembly

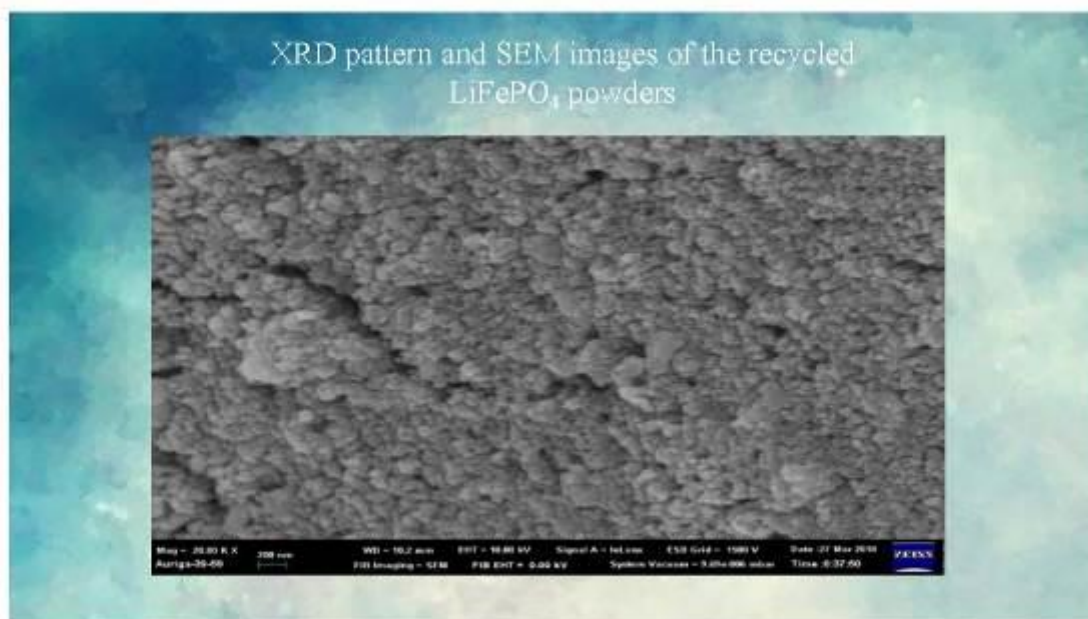


LiFePO_4 cathode

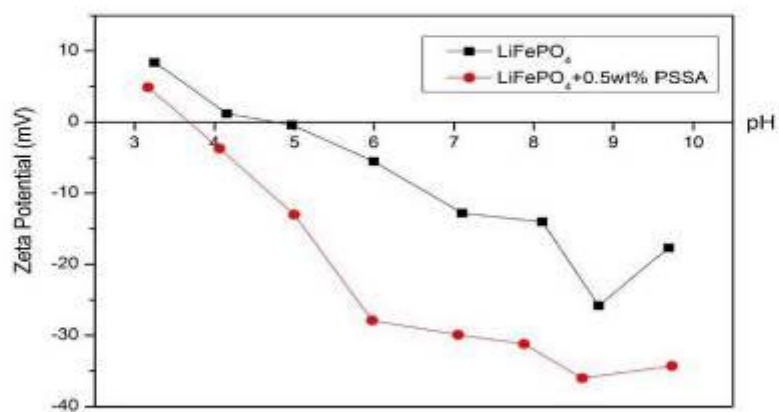


Graphite anode

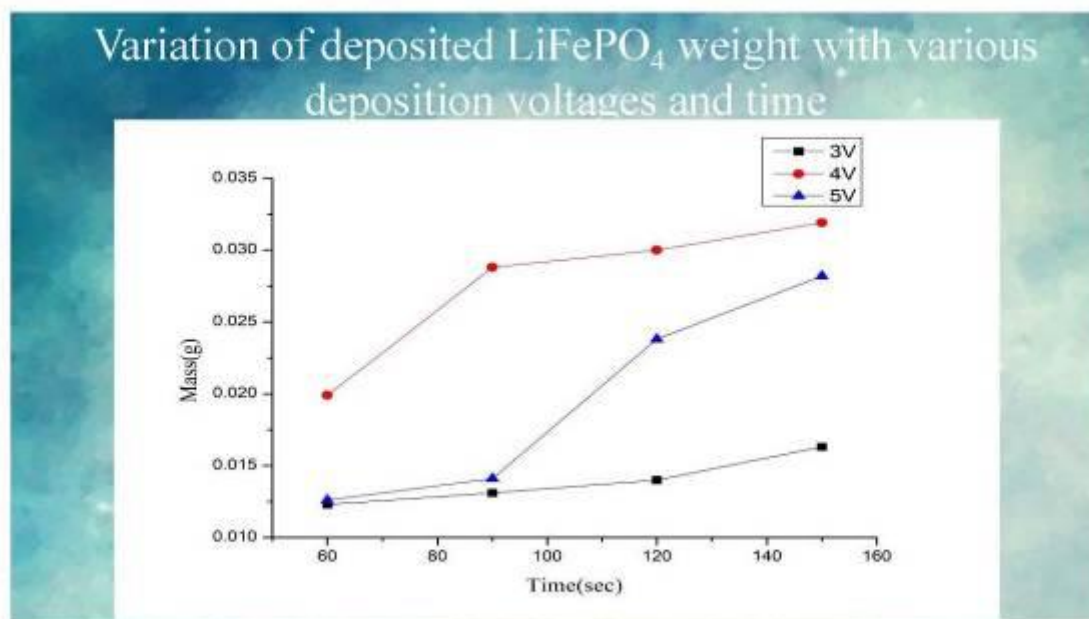




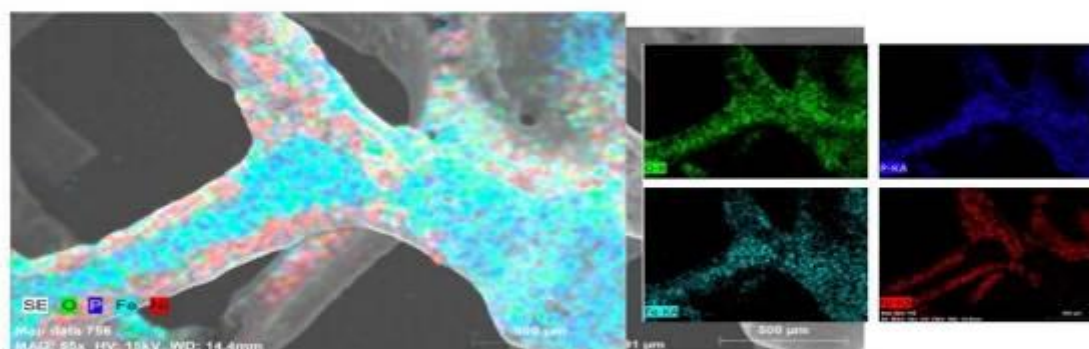
Zeta potential at different pH



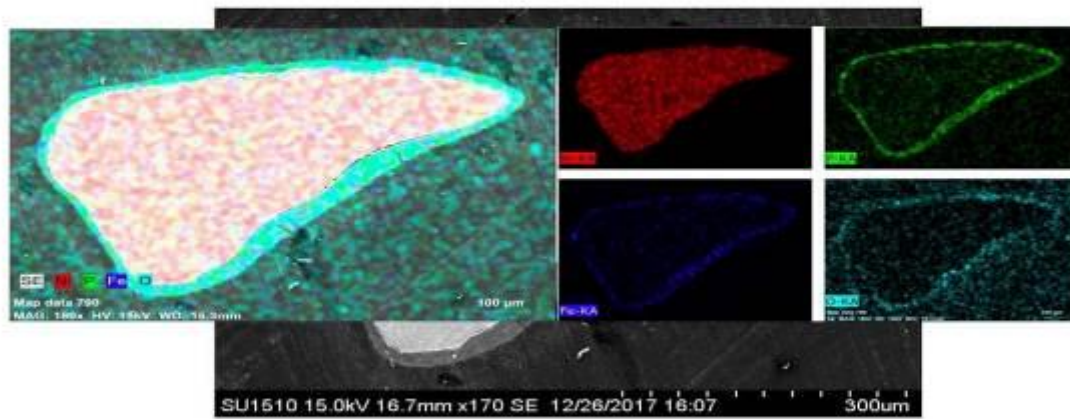
33



Microstructure after electrophoresis at 4 V for 2 min

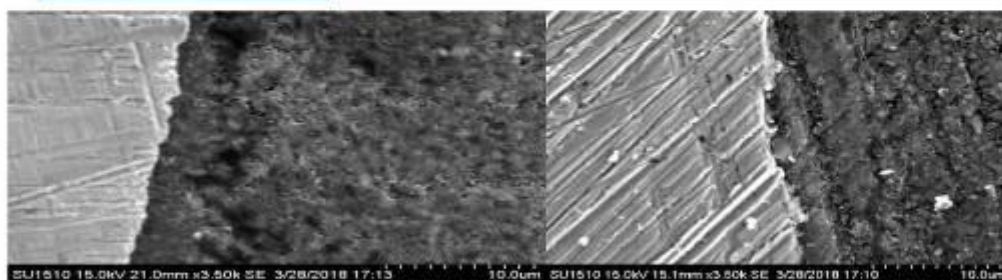


Microstructure after electrophoresis at 4 V for 2 min



Effect of heat-treatment on the peeling rate

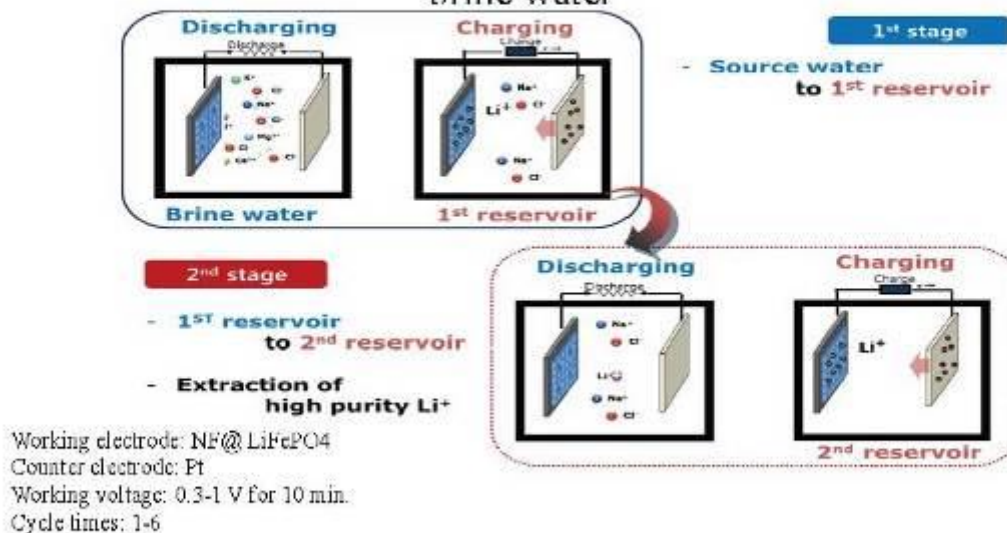
heat-treatment 300°C-1h	Before ultrasonic (g)	After ultrasonic (g)	peeling rate
after	0.5840	0.5830	0.17%
before	0.7773	0.7692	1.04%



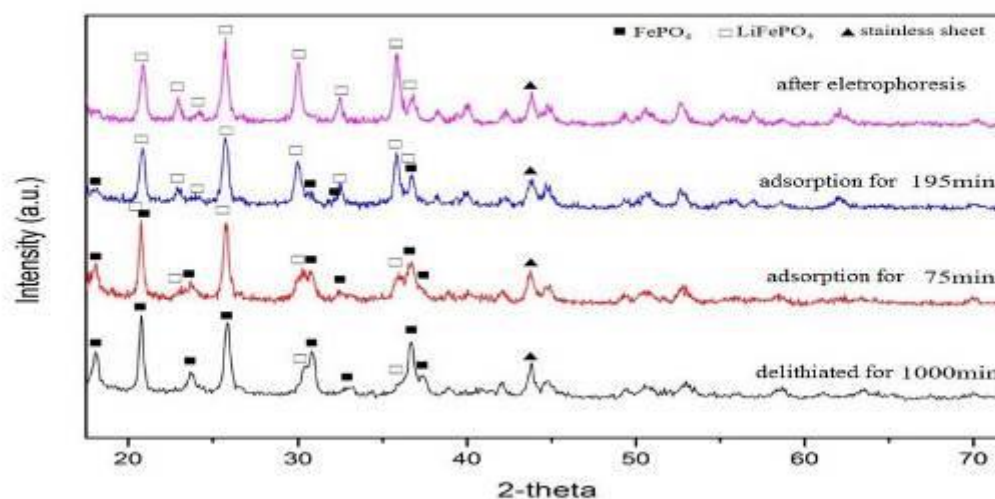
Before
ultrasonic

After ultrasonic

Schematic of lithium ion capturing process in simulated brine water

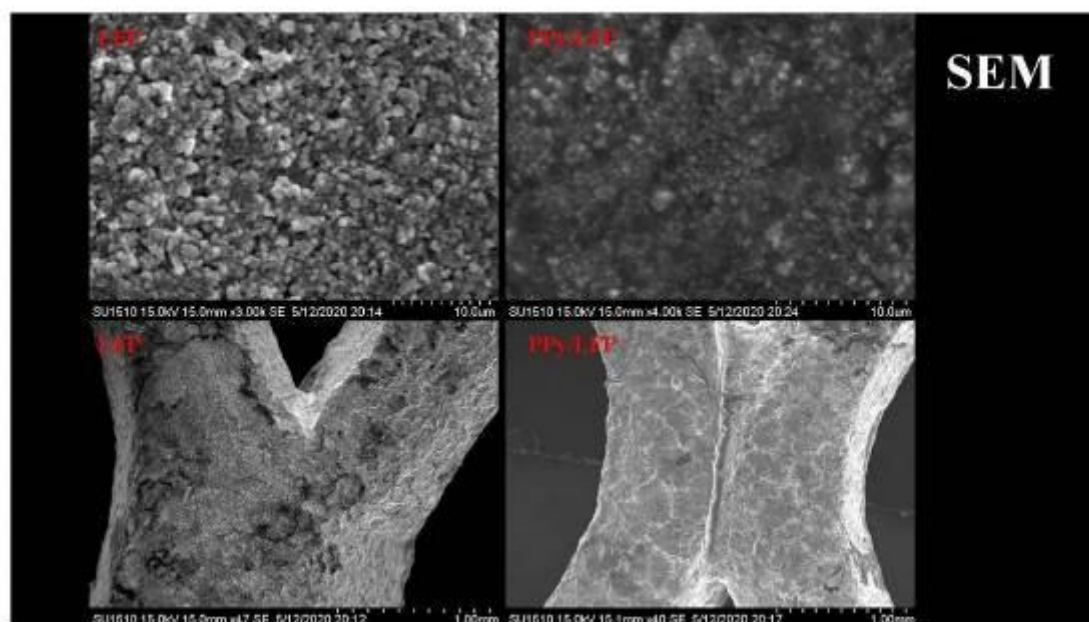
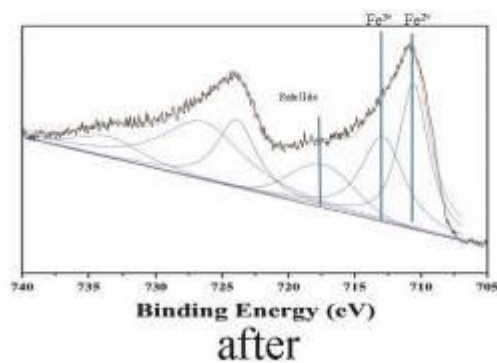
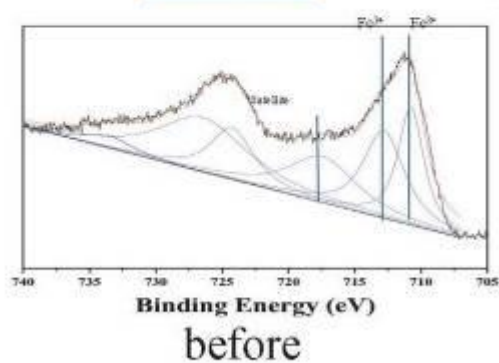


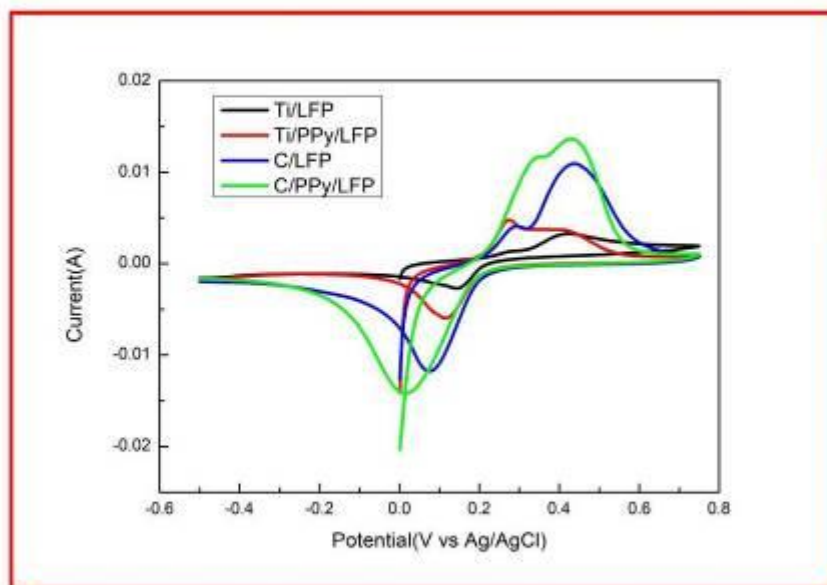
XRD patterns



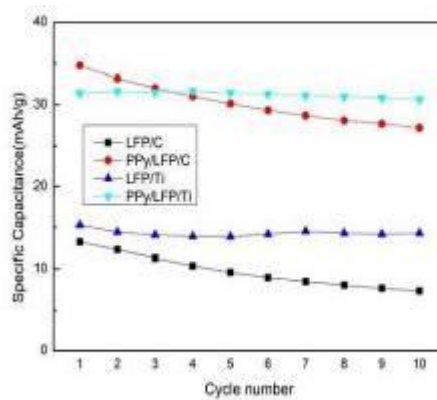
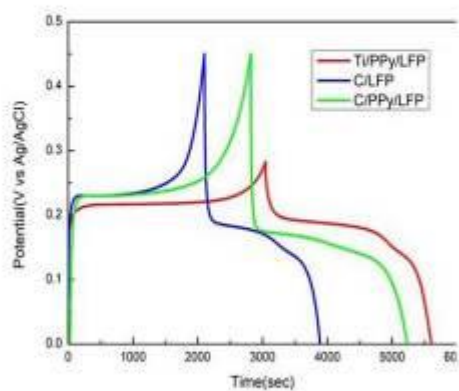
XPS of the sample before and after electro-adsorption

	Fe^{2+}	Fe^{3+}	$\text{Fe}^{2+}/\text{Fe}^{3+}$
before	10905.5	12826.11	0.8502
after	15199.03	12562.5	1.2098

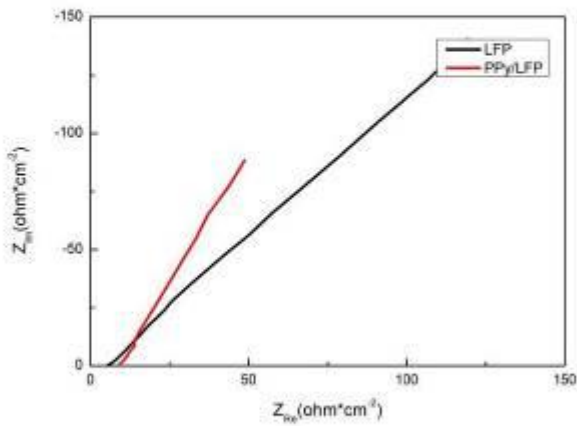




GCD



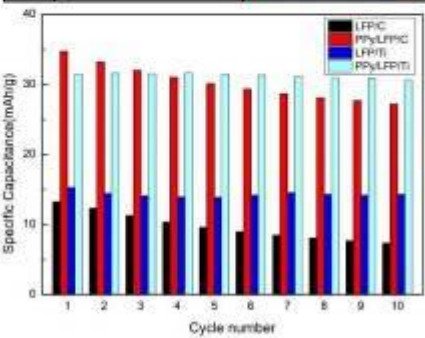
EIS



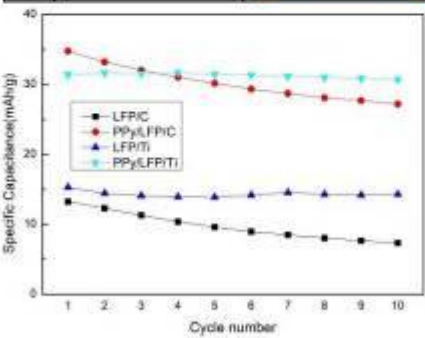
低頻區的斜直線對應離子在固態電極中的擴散阻抗，直線斜率越大表示離子擴散阻抗越小

多次GCD比較

	LFP	PPy/LFP
Ti	平均14.31mAh/g	平均31.26mAh/g
C	平均9.73mAh/g	平均30.2mAh/g

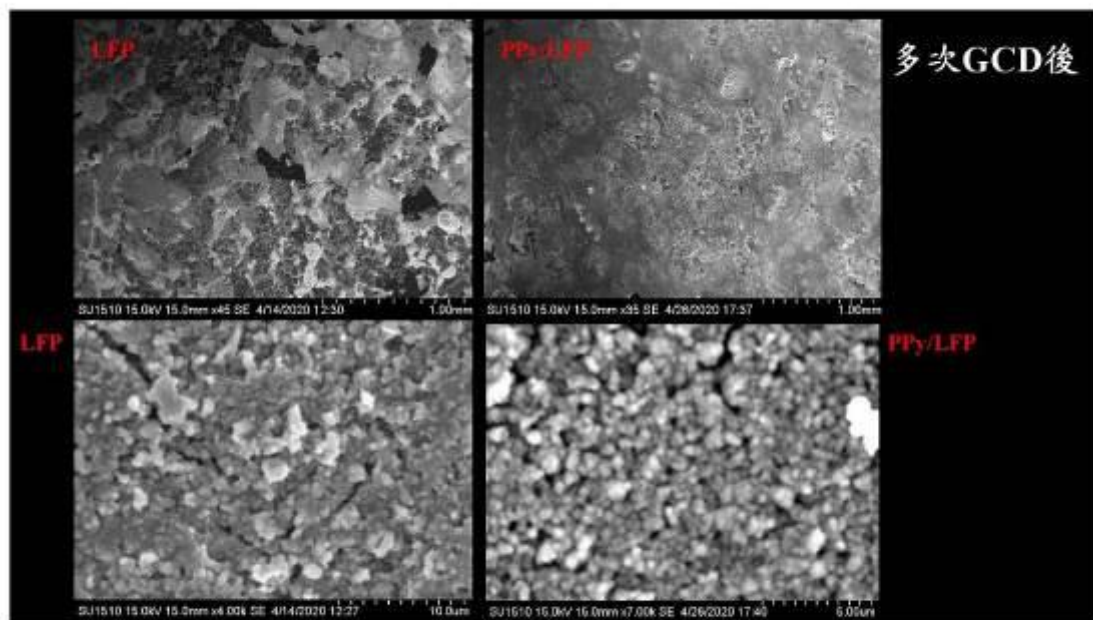


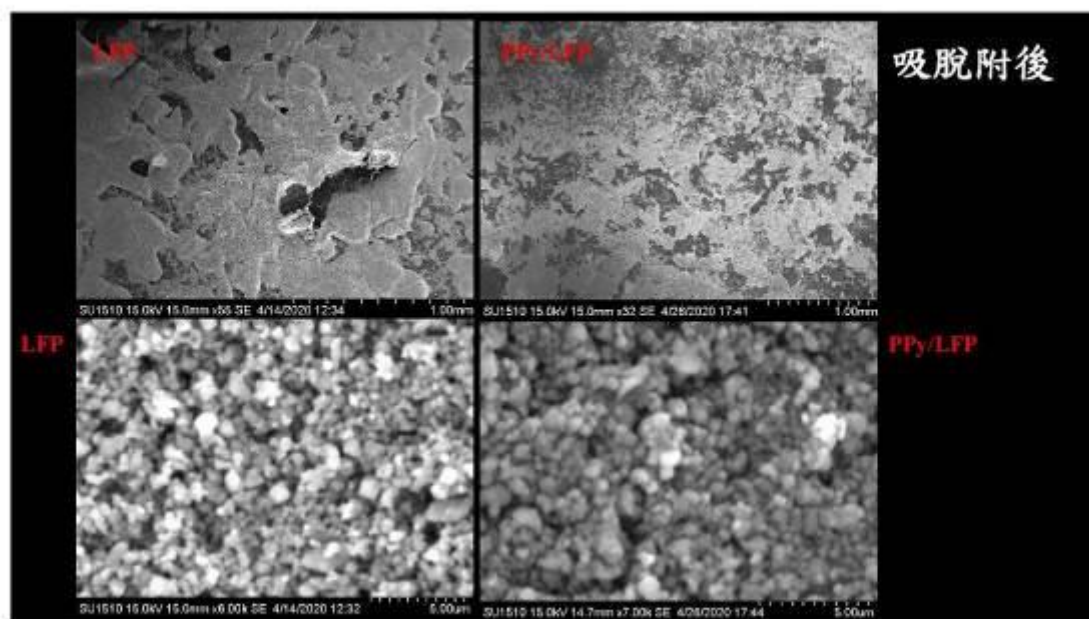
	LFP	PPy/LFP
Ti	在十次循環內仍保持較一致的利用率	在十次循環內仍保持較一致的利用率
C	衰退45%	衰退20%



多次GCD結果

1. PPy薄膜可以提高LFP薄膜的利用率，除了每克LFP電容量提高，也能利用更多LFP。
2. 鈦網為多孔材質，相比碳片，表面積更大，參與反應的LFP更容易觸碰到電解液，因此利用的每克LFP電容量提高，同時也較能維持利用率。
3. 導電高分子PPy+多孔材料鈦網，能賦予鋰離子篩更好的效率。

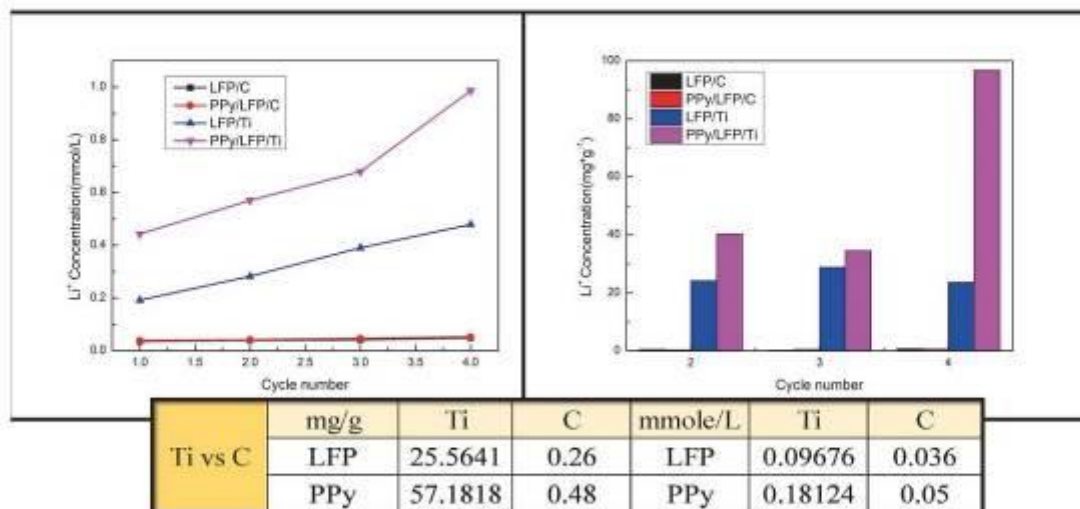




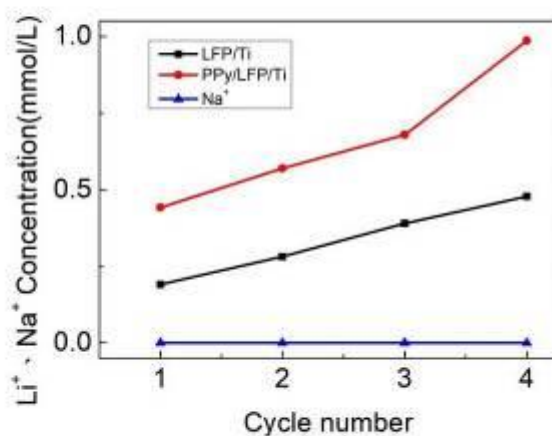
吸脫附實驗

- 吸附液: 30mM of each (NaCl, KCl, CaCl₂ and MgCl₂, LiCl) or 100mM LiCl
- 脫附液: 30mM LiCl
- 吸脫附時間: 30min
- 吸附電壓: 0.3V $e^- + Li^+ + FePO_4 \rightarrow LiFePO_4$
- 脫附電壓: 1.0V $LiFePO_4 \rightarrow Li^+ + FePO_4 + e^-$
- 次數: 4

Lithium adsorption for different cycles



Ion adsorption selectivity



通過ICP確認Na⁺
濃度不會改變



證明鋰離子篩吸
脫附選擇性



Conclusions



- Two main recovery resources of lithium :
 - Brine water
 - Spent lithium battery
- This lithium recovery system successfully demonstrated that lithium can be selectively recovered from brine water which contains various cations.
- The Li^+ adsorption efficiency of the nanostructured lithium ion sieve and electro-adsorption is better than the traditional ion sieves.
- Novel lithium recovery technology from brine water by using spent LiFePO_4 battery was successfully developed.

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Thank you for your attention



EMO環境光陶瓷實驗室

